

Practitioner's Docket No. HMN 2 0016**CHAPTER II**

Preliminary Classification:

Proposed Class:

Subclass:

NOTE: "All applicants are requested to include a preliminary classification on newly filed patent applications. The preliminary classification, preferably class and subclass designations, should be identified in the upper right-hand corner of the letter of transmittal accompanying the application papers, for example 'Proposed Class 2, subclass 129.' " M.P.E.P., § 601, 7th ed.

**TRANSMITTAL LETTER
TO THE UNITED STATES ELECTED OFFICE (EO/US)**

(ENTRY INTO U.S. NATIONAL PHASE UNDER CHAPTER II)

| | | |
|---|------------------------------------|------------------------------------|
| INTERNATIONAL APPLICATION NO. | INTERNATIONAL FILING DATE | PRIORITY DATE CLAIMED |
| <u>PCT/EP99/08667</u> | <u>11 November 1999 (11.11.99)</u> | <u>11 November 1998 (11.11.98)</u> |
| TITLE OF INVENTION | | |
| <u>METHOD FOR REPROCESSING WASTE OILS, BASE OILS OBTAINED ACCORDING TO SAID</u> | | |
| <u>APPLICANT(S)</u> <u>METHOD AND USE THEREOF</u> | | |
| <u>Joachim POHLER, Michael MODLER, Detlev BRUHNKE, Holger HINDENBERG</u> | | |

Box PCT

**Assistant Commissioner for Patents
Washington D.C. 20231**

ATTENTION: EO/US**CERTIFICATION UNDER 37 C.F.R. § 1.10***(Express Mail label number is **mandatory**.)(Express Mail certification is **optional**.)

I hereby certify that this Transmittal Letter and the papers indicated as being transmitted therewith is being deposited with the United States Postal Service on this date MAY 3, 2001, in an envelope as "Express Mail Post Office to Addressee" Mailing Label Number EL 852784402 US, addressed to the: Assistant Commissioner for Patents, Washington, D.C. 20231.

Georgen B. George

(type or print name of person mailing paper)

Georgen B. George

Signature of person mailing paper

WARNING: Certificate of mailing (first class) or facsimile transmission procedures of 37 C.F.R. § 1.8 cannot be used to obtain a date of mailing or transmission for this correspondence.

***WARNING:** Each paper or fee filed by "Express Mail" **must** have the number of the "Express Mail" mailing label placed thereon prior to mailing. 37 C.F.R. § 1.10(b).

"Since the filing of correspondence under § 1.10 without the Express Mail mailing label thereon is an oversight that can be avoided by the exercise of reasonable care, requests for waiver of this requirement will **not** be granted on petition." Notice of Oct. 24, 1996, 60 Fed. Reg. 56,439, at 56,442.

NOTE: To avoid abandonment of the application, the applicant shall furnish to the USPTO, not later than 20 months from the priority date: (1) a copy of the international application, unless it has been previously communicated by the International Bureau or unless it was originally filed in the USPTO; and (2) the basic national fee (see 37 C.F.R. § 1.492(a)). The 30-month time limit may not be extended. 37 C.F.R. § 1.495.

WARNING: Where the items are those which can be submitted to complete the entry of the international application into the national phase are subsequent to 30 months from the priority date the application is still considered to be in the international state and if mailing procedures are utilized to obtain a date the express mail procedure of 37 C.F.R. § 1.10 must be used (since international application papers are not covered by an ordinary certificate of mailing—See 37 C.F.R. § 1.8).

NOTE: Documents and fees must be clearly identified as a submission to enter the national state under 35 U.S.C. § 371 otherwise the submission will be considered as being made under 35 U.S.C. § 111. 37 C.F.R. § 1.494(f).

I. Applicant herewith submits to the United States Elected Office (EO/US) the following items under 35 U.S.C. § 371:

- a. This express request to immediately begin national examination procedures (35 U.S.C. § 371(f)).
- b. The U.S. National Fee (35 U.S.C. § 371(c)(1)) and other fees (37 C.F.R. § 1.492) as indicated below:

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09/831104

JC03 Rec'd PCT/PTL 03 MAY 2001

2. Fees

| CLAIMS FEE | (1) FOR | (2) NUMBER FILED | (3) NUMBER EXTRA | (4) RATE | (5) CALCULATIONS | |
|---------------------|---|------------------|------------------|-------------|------------------|-----------|
| | TOTAL CLAIMS | 13 - 20 = | 0 | × \$18.00 = | \$ 0 | |
| | INDEPENDENT CLAIMS | 1 - 3 = | 0 | × \$80.00 = | 0 | |
| | MULTIPLE DEPENDENT CLAIM(S) (if applicable) | | | | + \$270.00 | |
| BASIC FEE** | <input type="checkbox"/> U.S. PTO WAS INTERNATIONAL PRELIMINARY EXAMINATION AUTHORITY Where an International preliminary examination fee as set forth in § 1.482 has been paid on the international application to the U.S. PTO: <ul style="list-style-type: none"> <input type="checkbox"/> and the international preliminary examination report states that the criteria of novelty, inventive step (non-obviousness) and industrial activity, as defined in PCT Article 33(1) to (4) have been satisfied for all the claims presented in the application entering the national stage (37 C.F.R. § 1.492(a)(4)) \$100.00 <input type="checkbox"/> and the above requirements are not met (37 C.F.R. § 1.492(a)(1)) \$690.00 <input checked="" type="checkbox"/> U.S. PTO WAS NOT INTERNATIONAL PRELIMINARY EXAMINATION AUTHORITY Where no international preliminary examination fee as set forth in § 1.482 has been paid to the U.S. PTO, and payment of an international search fee as set forth in § 1.445(a)(2) to the U.S. PTO: <ul style="list-style-type: none"> <input type="checkbox"/> has been paid (37 C.F.R. § 1.492(a)(2)) \$710.00 <input type="checkbox"/> has not been paid (37 C.F.R. § 1.492(a)(3)) \$1000.00 <input checked="" type="checkbox"/> where a search report on the international application has been prepared by the European Patent Office or the Japanese Patent Office (37 C.F.R. § 1.492(a)(5)) \$860.00 | | | | 860.00 | |
| | Total of above Calculations | | | | = 860.00 | |
| SMALL ENTITY | Reduction by 1/2 for filing by small entity, if applicable. Affidavit must be filed also. (note 37 C.F.R. § 1.9, 1.27, 1.28) | | | | | |
| | Subtotal | | | | | 860.00 |
| | Total National Fee | | | | | \$ 860.00 |
| | Fee for recording the enclosed assignment document \$40.00 (37 C.F.R. § 1.21(h)). (See Item 13 below). See attached "ASSIGNMENT COVER SHEET". | | | | | |
| TOTAL | Total Fees enclosed | | | | | \$ 860.00 |

*See attached Preliminary Amendment Reducing the Number of Claims.

Attached is a check money order in the amount of \$ 860.00

Authorization is hereby made to charge the amount of \$ _____

to Deposit Account No. 06-0308

to Credit card as shown on the attached credit card information authorization form PTO-2038.

WARNING: Credit card information should not be included on this form as it may become public.

Charge any additional fees required by this paper or credit any overpayment in the manner authorized above.

A duplicate of this paper is attached.

****WARNING:** "To avoid abandonment of the application the applicant shall furnish to the United States Patent and Trademark Office not later than the expiration of 30 months from the priority date: *** (2) the basic national fee (see § 1.492(a)). The 30-month time limit may not be extended." 37 C.F.R. § 1.495(b).

WARNING: If the translation of the international application and/or the oath or declaration have not been submitted by the applicant within thirty (30) months from the priority date, such requirements may be met within a time period set by the Office. 37 C.F.R. § 1.495(b)(2). The payment of the surcharge set forth in § 1.492(e) is required as a condition for accepting the oath or declaration later than thirty (30) months after the priority date. The payment of the processing fee set forth in § 1.492(f) is required for acceptance of an English translation later than thirty (30) months after the priority date. Failure to comply with these requirements will result in abandonment of the application. The provisions of § 1.136 apply to the period which is set. Notice of Jan. 3, 1993, 1147 O.G. 29 to 40.

3. A copy of the International application as filed (35 U.S.C. § 371(c)(2)):

NOTE: Section 1.495 (b) was amended to require that the basic national fee and a copy of the international application must be filed with the Office by 30 months from the priority date to avoid abandonment. "The International Bureau normally provides the copy of the international application to the Office in accordance with PCT Article 20. At the same time, the International Bureau notifies applicant of the communication to the Office. In accordance with PCT Rule 47.1, that notice shall be accepted by all the designated offices as conclusive evidence that the communication has duly taken place. Thus, if the applicant desires to enter the national stage, the applicant normally need only check to be sure the notice from the International Bureau has been received and then pay the basic national fee by 30 months from the priority date." Notice of Jan. 7, 1993, 1147 O.G. 29 to 40, at 35-36. See item 14c below.

- is transmitted herewith.
- is not required, as the application was filed with the United States Receiving Office.
- has been transmitted
 - by the International Bureau.
Date of mailing of the application (from form PCT/1B/308):
18 May 2000
 - by applicant on _____. (Date)

4. A translation of the International application into the English language (35 U.S.C. § 371(c)(2)):

- is transmitted herewith.
- is not required as the application was filed in English.
- was previously transmitted by applicant on _____. (Date)
- will follow.

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5. Amendments to the claims of the International application under PCT Article 19 (35 U.S.C. § 371(c)(3)):

NOTE: *The Notice of January 7, 1993 points out that 37 C.F.R. § 1.495(a) was amended to clarify the existing and continuing practice that PCT Article 19 amendments must be submitted by 30 months from the priority date and this deadline may not be extended. The Notice further advises that: "The failure to do so will not result in loss of the subject matter of the PCT Article 19 amendments. Applicant may submit that subject matter in a preliminary amendment filed under section 1.121. In many cases, filing an amendment under section 1.121 is preferable since grammatical or idiomatic errors may be corrected." 1147 O.G. 29-40, at 36.*

a. are transmitted herewith.

b. have been transmitted

i. by the International Bureau.

Date of mailing of the amendment (from form PCT/1B/308):

ii. by applicant on _____. (Date)

c. have not been transmitted as

i. applicant chose not to make amendments under PCT Article 19.
Date of mailing of Search Report (from form PCT/ISA/210.):

25 Feb 2000.

ii. the time limit for the submission of amendments has not yet expired.
The amendments or a statement that amendments have not been made will be transmitted before the expiration of the time limit under PCT Rule 46.1.

6. A translation of the amendments to the claims under PCT Article 19 (38 U.S.C. § 371(c)(3)):

a. is transmitted herewith.

b. is not required as the amendments were made in the English language.

c. has not been transmitted for reasons indicated at point 5(c) above.

7. A copy of the international examination report (PCT/IPEA/409)

is transmitted herewith.

is not required as the application was filed with the United States Receiving Office.

8. Annex(es) to the international preliminary examination report

a. is/are transmitted herewith.

b. is/are not required as the application was filed with the United States Receiving Office.

9. A translation of the annexes to the international preliminary examination report

a. is transmitted herewith.

b. is not required as the annexes are in the English language.

10. An oath or declaration of the inventor (35 U.S.C. § 371(c)(4)) complying with 35 U.S.C. § 115

- was previously submitted by applicant on _____
Date
- is submitted herewith, and such oath or declaration
 - is attached to the application.
 - identifies the application and any amendments under PCT Article 19 that were transmitted as stated in points 3(b) or 3(c) and 5(b); and states that they were reviewed by the inventor as required by 37 C.F.R. § 1.70.
- will follow.

II. Other document(s) or information included:

11. An International Search Report (PCT/ISA/210) or Declaration under PCT Article 17(2)(a):

- is transmitted herewith.
- has been transmitted by the International Bureau.
Date of mailing (from form PCT/IB/308): 18 May 2000
- is not required, as the application was searched by the United States International Searching Authority.
- will be transmitted promptly upon request.
- has been submitted by applicant on _____
Date

12. An Information Disclosure Statement under 37 C.F.R. §§ 1.97 and 1.98:

- is transmitted herewith.
Also transmitted herewith is/are:
 - Form PTO-1449 (PTO/SB/08A and 08B).
 - Copies of citations listed.
- will be transmitted within THREE MONTHS of the date of submission of requirements under 35 U.S.C. § 371(c).
- was previously submitted by applicant on _____
Date

13. An assignment document is transmitted herewith for recording.

A separate "COVER SHEET FOR ASSIGNMENT (DOCUMENT) ACCOMPANYING NEW PATENT APPLICATION" or FORM PTO 1595 is also attached.

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14. Additional documents:

- a. Copy of request (PCT/RO/101)
- b. International Publication No. WO 00/27957
 - i. Specification, claims and drawing
 - ii. Front page only
- c. Preliminary amendment (37 C.F.R. § 1.121)
- d. Other

PCT/IB/301PCT/IB/304PCT/IB/33215. The above checked items are being transmitted

- a. before 30 months from any claimed priority date.
- b. after 30 months.

16. Certain requirements under 35 U.S.C. § 371 were previously submitted by the applicant on _____, namely:

AUTHORIZATION TO CHARGE ADDITIONAL FEES

WARNING: Accurately count claims, especially multiple dependant claims, to avoid unexpected high charges if extra claims are authorized.

NOTE: "A written request may be submitted in an application that is an authorization to treat any concurrent or future reply, requiring a petition for an extension of time under this paragraph for its timely submission, as incorporating a petition for extension of time for the appropriate length of time. An authorization to charge all required fees, fees under § 1.17, or all required extension of time fees will be treated as a constructive petition for an extension of time in any concurrent or future reply requiring a petition for an extension of time under this paragraph for its timely submission. Submission of the fee set forth in § 1.17(a) will also be treated as a constructive petition for an extension of time in any concurrent reply requiring a petition for an extension of time under this paragraph for its timely submission." 37 C.F.R. § 1.136(a)(3).

NOTE: "Amounts of twenty-five dollars or less will not be returned unless specifically requested within a reasonable time, nor will the payer be notified of such amounts; amounts over twenty-five dollars may be returned by check or, if requested, by credit to a deposit account." 37 C.F.R. § 1.26(a).

- Please charge, in the manner authorized above, the following additional fees that may be required by this paper and during the entire pendency of this application:
 - 37 C.F.R. § 1.492(a)(1), (2), (3), and (4) (filing fees)

WARNING: Because failure to pay the national fee within 30 months without extension (37 C.F.R. § 1.495(b)(2)) results in abandonment of the application, it would be best to always check the above box.

37 C.F.R. § 1.492(b), (c) and (d) (presentation of extra claims)

NOTE: Because additional fees for excess or multiple dependent claims not paid on filing or on later presentation must only be paid on these claims cancelled by amendment prior to the expiration of the time period set for response by the PTO in any notice of fee deficiency (37 C.F.R. § 1.492(d)), it might be best not to authorize the PTO to charge additional claim fees, except possible when dealing with amendments after final action.

37 C.F.R. § 1.17 (application processing fees)
 37 C.F.R. § 1.17(a)(1)–(5) (extension fees pursuant to § 1.136(a)).
 37 C.F.R. § 1.18 (issue fee at or before mailing of Notice of Allowance, pursuant to 37 C.F.R. § 1.311(b))

NOTE: Where an authorization to charge the issue fee to a deposit account has been filed before the mailing of a Notice of Allowance, the issue fee will be automatically charged to the deposit account at the time of mailing the notice of allowance. 37 C.F.R. § 1.311(b).

NOTE: 37 C.F.R. § 1.28(b) requires "Notification of any change in loss of entitlement to small entity status must be filed in the application . . . prior to paying, or at the time of paying . . . issue fee." From the wording of 37 C.F.R. § 1.28(b): (a) notification of change of status must be made even if the fee is paid as "other than a small entity" and (b) no notification is required if the change is to another small entity.

37 C.F.R. § 1.492(e) and (f) (surcharge fees for filing the declaration and/or filing an English translation of an International Application later than 30 months after the priority date).



SIGNATURE OF PRACTITIONER

Scott A. McCollister

(type or print name of practitioner)

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Customer No.:

(Transmittal Letter to the United States Elected Office (EO/US) [13-18]—page 8 of 8)

PATENT

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

IN RE APPLICATION OF : Joachim Pöhler, et al.
FOR : **METHOD FOR REPROCESSING WASTE
OILS, BASE OILS OBTAINED ACCORDING
TO SAID METHOD AND USE THEREOF**
SERIAL NO. : Unknown
FILED : Herewith
ATTORNEY DOCKET NO.: HMN 2 0016
Cleveland, Ohio 44114-2518
May 3, 2001

PRELIMINARY AMENDMENT

Assistant Commissioner For Patents
Washington, D.C. 20231

Dear Sir:

Prior to calculation of the filing fee and substantive examination of the above-referenced patent application, Applicants respectfully request amendment of the application as follows. A clean copy of the claims appears below and a marked-up version is attached as an appendix.

IN THE CLAIMS:

1. Method for reprocessing waste oils and production of high-grade base oils, comprising the following steps:

A) distillation of the waste oil for removal of low boiling organic fractions and removal of water;

B) distillation under vacuum of the waste oil obtained according to step A) for segregation of fuel oil and diesel fractions with boiling cut of about 170 to 385°C in form of high grade fuel oils;

C) non-destructive distillation of the distillation residue from step B) by means of thin film evaporation in high vacuum to obtain a lubricating oil fraction with a standard viscosity range, which can be separated, as needed, via a subsequent distillative fractioning step, optionally under vacuum, into boiling cuts of different viscosity states;

D) if appropriate, non-destructive distillation of the bottom product from step C) in order to obtain a lubricating oil fraction of higher viscosity state from the higher boiling range which can be segregated, as needed, optionally under vacuum, by means of a subsequent distillative fractioning step;

E) extraction of the fraction or fractions in the form of lubricating oil fractions or boiling cuts of different viscosity states from step C) and optionally D) with N-methyl-2-pyrrolidone (NMP) and/or N-formylmorpholine (NMF) as extraction medium in order to obtain very high grade base oils whereby the extraction is executed in such manner that undesirable constituents are removed in an almost quantitative manner, and the contents of the polycyclic aromatic hydrocarbons (PAK) and polychlorinated biphenylenes (PCB) lies, respectively, below 1 mg/kg.

2. Method according to Claim 1, wherein the waste oils to be reprocessed are treated with concentrated watery alkaline solution as reagent in order to segregate interfering constituents.

3. Method according to Claim 2, wherein the alkaline solution is added during distillation according to step A).

4. Method according to Claim 2 wherein the alkaline solution is potassium hydroxide solution.

5. Method according to Claim 4, wherein the alkaline solution is approximately 5 to 50% potassium hydroxide solution.

6. Method according to Claim 5, wherein the feed (charge for extraction) is imparted an alkalinity reserve, which prevents the otherwise usual, partially even irreversible, acidulation of the reclaimed extraction medium.

7. Method according to Claim 1 wherein the distillation in step A) is performed at normal pressure or at slight under pressure of up to approximately 600 mbar and at a temperature of approximately 140 to 150°C.

8. Method according to claim 1 wherein the extraction is performed in an extraction column, by counter flow process.

9. Method according to claim 1 wherein the extraction is performed in isothermal manner, at a temperature in the range of approximately 50 to 90°C.

10. Method according to Claim 9, wherein an extract phase is cooled down and that the settling oil phase is again added to the feed.

11. Method according to claim 1 wherein the extraction is performed with a temperature gradient, whereby the temperature is adjusted at the column head (run off raffinate) to approximately 50 to 90°C and at the column end (extract run-off) to approximately 10 to 50°C.

12. Method according to claim 1 wherein the waste oil to be reprocessed has a contents of polychlorinated biphenyls (PCB) or PCB substitutes of up to approximately 250 mg/kg.

13. Method according to claim 1 wherein the waste oil to be reprocessed has a contents of vegetable oil(s) of up to approximately 5%.

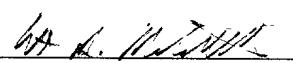
Please cancel claims 14 and 15.

Remarks

Applicants respectfully request that the foregoing amendments be entered prior to substantive examination of the application. These changes are submitted to eliminate the multiple dependent claims in the application.

Respectfully submitted,
FAY, SHARPE, FAGAN
MINNICH & McKEE, LLP

Date: May 3, 2001


Scott A. McCollister
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(216) 861-5582

CERTIFICATE OF MAILING

I hereby certify that this **PRELIMINARY AMENDMENT** is being deposited with the United States Postal Service as **EXPRESS MAIL** in an envelope numbered **EL 852784402 US** addressed to Assistant Commissioner for Patents, Washington, D.C. 20231, on May 3, 2001.

By: 
Georgette B. George

VERSION WITH MARKINGS TO SHOW CHANGES MADE

1. (Amended) Method for reprocessing waste oils and production of high-grade base oils, comprising the following steps:

A) distillation of the waste oil for removal of low boiling organic fractions[, as well as drying of waste oil by] and removal of water;

B) distillation under vacuum of the waste oil obtained according to step A) for segregation of fuel oil and diesel fractions with boiling cut of [approximately] about 170 to 385°C in form of high grade fuel oils;

C) non-destructive distillation of the distillation residue from step B) by means of thin film evaporation in high vacuum to obtain a lubricating oil fraction with a standard viscosity range, which can be separated, as needed, via a subsequent distillative fractioning step [- if appropriate under vacuum -] optionally under vacuum, into boiling cuts of different viscosity states;

D) if appropriate, non-destructive distillation of the bottom product from step C) in order to obtain a lubricating oil fraction of higher viscosity state from the higher boiling range which can be segregated, as needed, [possible under vacuum] optionally under vacuum, by means of a subsequent distillative fractioning step;

E) extraction of the fraction or fractions in the form of lubricating oil fractions or boiling cuts of different viscosity states from step C) and optionally D) with N-methyl-2-pyrrolidone (NMP) and/or N-formylmorpholine (NMF) as extraction medium in order to obtain very high grade base oils whereby the extraction is executed in such manner that undesirable constituents are removed in an almost quantitative manner, and the contents of the polycyclic aromatic hydrocarbons (PAK) and polychlorinated biphenyls (PCB) lies, respectively, [clearly] below 1 mg/kg.

2. (Amended) Method according to Claim 1, [characterized in that] wherein the waste oils to be reprocessed are treated with concentrated watery alkaline solution as reagent in order to segregate interfering constituents[, in particular for process step C)].

3. (Amended) Method according to Claim 2, [characterized in that] wherein the alkaline solution is added during distillation according to step A).

4. (Amended) Method according to Claim 2 [or 3, characterized in that] wherein the alkaline solution is potassium hydroxide solution.

5. (Amended) Method according to Claim 4, [characterized in that] wherein the alkaline solution is [highly concentrated specifically] approximately 5 to 50% potassium hydroxide solution.

6. (Amended) Method according to Claim 5, [characterized in that] wherein the feed (charge for extraction) is imparted an alkalinity reserve, which prevents the otherwise usual, partially even irreversible, acidulation of the reclaimed extraction medium.

7. (Amended) Method according to Claim 1 [or 2, characterized in that] wherein the distillation in step A) is performed at normal pressure or at slight under pressure of up to approximately 600 mbar and at a temperature of approximately 140 to 150°C.

8. (Amended) Method according to [at least one of the preceding Claims, characterized in that] claim 1 wherein the extraction is performed in an extraction column, by counter flow process.

9. (Amended) Method according to [at least one of the preceding claims, characterized in that] claim 1 wherein the extraction is performed in isothermal manner, at a temperature in the range of approximately 50 to 90°C.

10. (Amended) Method according to Claim 9, [characterized in that the] wherein an extract phase is cooled down and that the settling oil phase is again added to the feed.

11. (Amended) Method according to [at least one of Claims 1 to 8, characterized in that] claim 1 wherein the extraction is performed with a temperature gradient, whereby the temperature is adjusted at the column head (run off raffinate) to

approximately 50 to 90°C and at the column end (extract run-off) to approximately 10 to 50°C.

12. (Amended) Method according to [at least one of the preceding Claims, characterized in that] claim 1 wherein the waste oil to be reprocessed has a contents of polychlorinated biphenylenes (PCB) or PCB substitutes of up to approximately 250 mg/kg.

13. (Amended) Method according to [at least one of the preceding claims, characterized in that] claim 1 wherein the waste oil to be reprocessed has a contents of vegetable oil(s) of up to approximately 5%.

**METHOD FOR REPROCESSING WASTE OILS, BASE OILS
OBTAINED ACCORDING TO SAID METHOD AND USE THEREOF**

The present invention relates to a method for reprocessing waste oils, whereby waste oil is treated by means of distillation and extraction method, base oils obtained according to said method and their use thereof.

These days, disposal of waste products is becoming increasingly more important, in particular the elimination of environmentally noxious substances, such as halogenated hydrocarbons, aromatic compounds and similar. These represent acute and latent risks to human health and to other environmental resources, such as water, soil, air, plants and animals. In order to evaluate the actual risk potentials, it is necessary to examine and appraise material compounds and their concentrations. Such data serve as basis for type and scope of the disposal measures that need to be taken.

Of immense interest is hereby the disposal of products burdened with noxious substances, which occur in larger volume, such as waste oils, for example. Disposing these can be done either by elimination of the loaded products or by utilization of same, whereby utilization is basically to be preferred.

Utilization is generally possible via two routes: the product can be materially utilized, i.e., it can be reprocessed, or it can be utilized energetically as fuel. Certain criteria must be observed in such endeavor, which depend upon the product involved and the respective noxious matter burden.

Waste oils, for example, are subject to the so-called Waste Oil Ordinance (AltölV) of October 27, 1987, which regulates the reprocessing, registration, identification, collection and disposal of waste oils. Waste oils to be reprocessed are, as a rule, prohibited

from exceeding a maximal value of 20 ppm of polychlorinated biphenylene (PCB) and a total halogen content of 0.2%. Exceptions, however, are quite possible, depending upon the chosen reprocessing method. Several methods for reprocessing of waste oils or of used oils are known in the state of the art. U.S. Patent 4,021,333 from the year 1977 describes, 5 for example, a method for reprocessing waste or used oils, which includes the following steps:

A) Distillation of the oil for eliminating a pre-fractionation with a viscosity essentially below that of lubricating oil and a flash point below 121°C, ascertained according to the Tag or Pensky-Martens method;

10 B) Continued distillation in order to obtain a distillate having, in essence, the viscosity of lubricating oil;

C) Extraction of impurities from the distillate of step B) with an organic, liquid extraction agent, which is essentially non-mixable with the distillate, and

15 D) Segregation from the distillate of the organic solvent and the impurities dissolved therein.

Named as organic extraction agents, are in particular: ethanol, diacetone-alcohol, ethylene-glycol-mono(low-alkyl)ether, diethylene-glycol, diethylene-glycolmono(low-alkyl)ether, o-chlorophenol, furfural, acetone, formic acid, 4-butyrolactone, low-alkyl-ester of low mono-and dicarboxylic acids, dimethylformamide, 2-pyrrolidone and N-(low-alkyl)2-pyrrolidone, epi-chlorohydrin, dioxane, morpholine, low-alkyl-and amino(low-alkyl)morpholine, benzonitrile and di-(low-alkyl)sulfoxide and phosphonate. Preferred extraction agents are ethylenglycol-monomethyl-ether, dimethylformamid or N-methyl-2-pyrrolidone. In addition, in step C), 20 to 50 parts by weight of extraction agents are employed for 100 parts of weight of obtained distillate from 20 step B).

Distillation is to be performed without fractionating column or similar equipment. It is possible to remove, in a prior step, bothersome components, by means of a thinning agent, in form or an organic solvent, which step, additionally, may be preceded by heating the waste oil with a watery, highly alkaline solution. This method, however, 5 does not always produce satisfactory results with respect to the quality of the reprocessed oils, which contain, as before, high loads of noxious substances after the reprocessing.

The present invention is therefore based on the object of further improving the initially described method so that the obtained base oils have the lowest possible load of noxious matter, such as aromatic compounds and specifically polycyclic aromatic 10 hydrocarbons. In addition, design of method control and potential starter materials should be flexible. At the same time, notwithstanding high quality of the obtainable base oils, good yields should be attained.

According to the invention, the above object is solved by a method for reprocessing of waste oils and production of high grade base oils comprising the following 15 steps:

- A) Distillation of the waste oil for removal of low-boiling organic fractions, as well as drying of the waste oil by removal of water;
- B) Distillation of waste oil obtained according to step A) under vacuum for separation of fuel oil and diesel fractions, with boiling cut of approximately 170 to 20 385 °C, in form of high-grade heating fuels;
- C) Non-destructive distillation of the distillation residue from step B) by means of thin-film evaporation in high vacuum for obtaining a lubricating oil fraction with a standard viscosity range, which may be followed, if needed, by a subsequent distillative fractioning step, possibly under vacuum, which can be divided into boiling 25 fractions of different viscosity states;

D) If applicable, non-destructive distillation of the bottom product from step C) for obtaining a lubricating oil fraction of higher viscosity state from the higher boiling range, which, depending upon requirement, can be divided into a subsequent distillative fractionating step, possibly under vacuum; and

5 E) Extraction of fraction or fractions in the form of lubricating oil fractions or boiling cuts of different viscosity from step C) and, optionally from step D) with N-methyl-2-pyrrolidone (NMP) and/or N-formylmorpholine (NMF) as extraction agent for obtaining extremely high grade base oils, whereby the extraction is undertaken in such manner that undesirable constituents are removed in almost quantitative manner
10 and the contents of polycyclic aromatic hydrocarbons (PAK) and polychlorinated biphenylenes (PCB) respectively, is clearly below 1mg/kg.

15 The term "waste oil" in the present invention shall have the meaning of any used semi-liquid or liquid substance, which is comprised in total or in part of mineral oils or synthetic oils, as well as any oil containing residue, including water-oil mixtures or similar. Thus, it is possible to use all waste oils suitable for reprocessing into lubricating oils, in particular used combustion engine and transmission oils, mineral machine oils, turbine oils and hydraulic oils, including their synthetic and semi-synthetic constituents or mineral hydrocarbon basis.

20 The inventive method for reprocessing said waste oils is described in detail below:

In a first step A), water fractions as well as fractions of low boiling constituents are distilled off, such as fractions of gasoline or solvents. This is preferably done at normal pressure or under light vacuum (up to approximately 600 mbar) at a temperature of approximately 140 to 150°C.

25 It is possible to facultatively employ in the inventive method, in particularly

beneficial manner, a concentrated watery potassium hydroxide solution. Said solution is already employed in this first step in order to avoid, during subsequent distillation steps, additionally burdening generation of the vacuum with water to be separated from the potassium hydroxide solution. The potassium hydroxide solution is preferably highly 5 concentrated, specifically approximately 5 to 50%.

At the same time one obtains, by means of the dehydration process executed in the initial step A), the concentration of the potassium hydroxide as an extremely homogeneously distributed, highly concentrated and therefore highly effective acting reagent for binding acid constituents in the charged waste oil, including extensive 10 demetallization of the charged waste oils. Furthermore, the use of potassium hydroxide solution, which causes formation of specific "soaps", produces in the subsequently described thin film evaporation in step C), a particularly free-flowing and homogenous distillation residue. In contrast thereto, other alkalies, such as sodium compounds described in US-PS 4,021,333, for example, tend to produce precipitations and 15 agglomerations, which can significantly interfere with the further process sequence. By utilizing potassium hydroxide solution it is possible to further improve the purity of the lubricating oil distillates and during the subsequent extraction, additional benefits are obtained with respect to process mode and chemical effect. In addition, by way of this type of alkaline treatment, it is possible to do away with a further step for mechanical separation 20 of solid precipitations.

After separation of water and solvents, the obtained fuel oil and diesel fractions with boiling average of approximately 170 to 385°C are removed from the waste oil in step B) by distillation in vacuum. The thus obtained residue is subjected, according to step C) to non-destructive thin film evaporation in high vacuum, in which one obtains 25 the lubricating oil fraction proper. For realizing desired viscosity states, the latter can subsequently be fractionated once again.

The residue from the thin film evaporation (bottom product) still contains

highly viscous and very valuable lubricating oil constituents which can be obtained in step D) and also fractionated, if applicable, by non-destructive distillation, such as subsequently added second thin film evaporation, with correspondingly higher distillation temperatures or lower pressure. Needless to say, step D) is not always necessary, but if used, will 5 improve the yield of base oil and thereby the economic efficiency of the process.

The lubricating oil fractions produced from the above described and (if applicable) fractionating steps (step C) and D)), are subsequently extracted with N-methyl-2-pyrrolidone (hereinafter identified as NMP), whereby qualitatively very high grade base oils are obtained for the production of lubricants. N-formylmorpholine (hereinafter 10 identified as NMF) has proven itself as particularly suitable as an alternative extraction medium, under the same conditions and with results comparable to the extraction with NMP. Of course, the lubricating oils or fractions from step C) and D) can, individually, be further processed or also partially added to each other and then processed further.

Extraction can preferably be done in a column (screen bottom, filling body), 15 preferably with counter-flow process. The use of filling bodies affords high soundness regarding process control and presents benefits relative to potential through-puts (volume flows) and extraction agent distribution of NMP or NMP in the oil. The ratio of NMP/oil or NMF/oil ranges between 0.5 and 2.0 (v/v) depending upon quality requirements of the base oils to be produced.

In contrast to standard practice, the use of NMP and/or NMP as continuous 20 phase, surprisingly, proved detrimental, inasmuch as unstable conditions set in very quickly, inside the column. The extraction agent must therefore be selected as disperse phase.

The extraction can basically be performed within a temperature range of 25 approximately 20 to 90°C.

The process method can be used with employment of a temperature gradient in the column. Temperatures ideally range between approximately 50 to 90°C at the column head (run off raffinate) and approximately 10 to 50°C at the column bottom (extract run off). Higher selectivity regarding the extraction agent is of benefit in this case 5 (for example NMP) with lower temperatures, so that base oil constituents dissolved in the extraction medium are re-dissolved, while the undesirable, to be removed, constituents remain dissolved. As a result, it is possible to attain a significantly higher yield of refined base oil.

Excellent results can also be achieved with a process mode using 10 unchanging temperature over the entire column path (isotherm). The optimal temperature range lies between approximately 50 to 90°C; depending upon requirements as to yield and quality, other ranges, however, are also possible. This is in contrast to the customary process method of first refining of paraffin-base crude oil distillates, where still high 15 paraffin fractions exist in the components to be extracted, which may already lead to precipitations at lower temperatures (<40°C), so that extractions must basically be performed at temperatures between 60 to 75°C. In the interest of economic yields, temperatures higher than 75°C are, in principle, not employed in crude oil distillate extraction. It is, however, possible to compensate for the drawback of isothermal operation 20 in that, vis-a-vis the process variation with temperature gradients, a lower yield is realized, inasmuch as there is no re-dissolution of the raffinate, as already described.

One proceeds as follows: the extraction phase is cooled down and the thereby segregating oil phase with lower extraction medium contents is again put into the oil feed of the column. This can also be called “external feedback”.

The NMP and/or NMF existing in the raffinate phase and the extract can be 25 reclaimed in standard method via succeeding distillation processes and returned to the process. As a result of the performed concentration of the potassium hydroxide solution in step A), an alkalinity reserve for extraction is established in the oil feed, which prevents

the otherwise partly irreversible formation of acid reaction products of the extraction medium.

According to this method one obtains, as a result, very high-grade base oils, whereby undesirable constituents are removed in an almost quantitative manner, i.e., the 5 contents of polycyclic aromatic hydrocarbons (abbreviated PAK) and of polychlorinated biphenylenes (abbreviated PCB) lies respectively clearly below 1 mg/kg.

The base oils are obtainable according to the above described method are also the object of the invention. Depending upon viscosity state of the distillate fractions produced according to step C) and step D), base oil qualities according to ASTM are 10 obtained with color value between 0.5 and 3.0. The neutralizing figure (abbreviated NZ) as measure of acid residual components in the base oil lies between 0.01 and 0.03 mg/KOH/g.

In contrast to the employed lubricating oil distillates according to step C) or step D), due to the extraction, increase occurs in the viscosity index (abbreviates VI) by 15 6 to 10 points. It is noteworthy in this respect that the viscosity index of the lubricating oil distillates is clearly higher than that of the usual first raffinate base oils, due to the existing synthetic oil fractions (polyalpha-olefins (PAO), hydro-crack oil (HC oils)).

By means of the inventive process, the aromatic fraction (abbreviated CA) in the base oil is clearly lowered. In particular, polycyclic aromatic hydrocarbons (PAK) 20 are removed in an almost quantitative manner (sum of PAK according to Grimmer - sum of a specified number of individual substances << 1 mg/kg, benzopyrene << 0.1 mg/kg). These are formed in part during utilization of the lubricating oils, in particular during the combustion processes in passenger car/truck engines and they play a significant role due to their health endangering properties, i.e. their cancer causing effect. This is particularly 25 the case with respect to benzopyrene, which is regarded as the principal PAK substance and which was included in the Ordinance of Hazardous Substances (GefStoffV). The

contents of these compounds in oil components and oil preparations will clearly be assigned greater significance in future public debate.

5 Among the currently known methods for reprocessing waste oils into base oils, there is presently no method that is capable of removing the PAK to the extent as it is possible with the method or combinations of the method according to the invention.

Surprisingly, not only are the already earlier mentioned waste oils being considered for reprocessing according to the method, tests have shown that highly contaminated waste oils can also be employed. Thus, it is also possible to reprocess oil burdened with polychlorinated biphenylene (PCB) or PCB-substitutes, with contents of up 10 to 50 mg/kg according to DIN 51527-I (Total contents according to LAGA 250 mg/kg, [LAGA = Country Working Group Waste]. Polychlorinated biphenylenes are a group of compounds with different toxicities depending upon chlorination contents. They are classified, based on documented suspicion, according to their cancer causing potential (MAK: appendix IIIB), as a result of which segregation of these compounds is required 15 from a toxicological aspect.

Waste oils reprocessed according to the inventive method with the above mentioned PCB contents present a PCB contents in the base oils obtained according to the invention which lies below the detection limits of the analytical method. This is of importance to the extent that both National as well as European regulations with respect 20 to reprocessing of such burdened waste oils will explicitly allow such processes only in individual instances when high qualitative standards are reached for the obtained base oil. The inventive method fulfills these requirements.

In addition, waste oils can also be reprocessed with a content of vegetable oils, so called readily decomposable oils. Up to approximately 5% of such oils can be 25 contained in the waste oil without detrimentally affecting the quality of the base oil.

Needless to say, the obtained base oils have multiple applications, for example as starter products for lubricants or for products in the petrochemical field, inasmuch as - based on the excellent quality - no restrictions exist within the scope of the invention.

5 The benefits related to the invention are multi-layered. The inventive method is far superior to the state of the art of standard processes of clay treatment, chemical treatment or hydration, as well as the known distillation methods. The inventive method can be performed without waste, since the extraction media NMP or NMF can be reclaimed and employed again, and the extract is utilized as fuel oil or fuel oil equivalent.
10 In contrast thereto, with the clay treatment method oil contaminated bleached earth remains behind, and with the hydration it becomes necessary to dispose of the spent catalysts, and the reaction gases (H₂S, HCl) need to be rendered harmless.

15 The energy balance according to the invention specific method is very favorable. It is possible to operate almost without pressure. For overcoming interior fluid friction and pipe line resistances during transport, pressures are only needed in the range of max.5 bar. The maximum temperature range lies at 230° in order to guarantee recuperation of the extraction medium for renewed use. In other processes, raffination effects set in only after temperatures have been reached between 290 and 300°C (Clay treatment), or it is necessary to employ, in addition, high pressures (hydration: 20 temperatures up to 350°C and operating pressures between 30 and 200 bar).

25 The inventive method also affords benefits with respect to operating safety, since the extraction media NMP or NMF are classified as non-toxic (classified as Xi: irritating according to Toxic Matter Ordinance, Risk Category A III, WGK 1). With standard hydration methods, on the other hand, hydrogen, being an easily flammable gas, demands high safety requirements. In addition, H₂S is formed as a highly toxic gas as well as hydrochloric acid as a highly corrosive gas.

Of special significance is the base oil quality achievable with the invention, as it is obtained via the NMP - or NMF extraction. The base oils have extraordinarily good color values, a low neutralizing figure (NZ) and a high viscosity index (VI). As a result of the inventive method, the aromatic fraction in the base oil is clearly reduced. 5 Specifically, polycyclic aromatic hydrocarbons (PAK) are removed in an almost quantitative manner (total PAK according to Grimmer << 1 mg/kg, benzopyrene <<0.1 mg./kg). The contents of polychlorinated biphenylene (PCB) also lies below the limit of detection in the obtained base oil.

With respect to the waste oils to be reprocessed, there are almost no 10 restrictions. It is possible to reprocess oil burdened with polychlorinated biphenylene (PCB) or PCB substitutes, with contents of up to 50 mg/kg according to DIN 51527-1 (total contents according to LAGA 250 mg/kg). The PCB contents in the base oils obtained according to the invention also lies in this instance below the detection limit of the 15 analytical method, i.e. strict National and European Regulations concerning reprocessing of waste oils are observed. In addition, contents of vegetable, biologically readily decomposable oils of up to 5% may be present in the waste oil to be reprocessed, without resulting in any detrimental influence upon the quality of the base oil. With the state of the art methods, it is impossible to achieve said outstanding quality of the base oil according to the invention. For example, with the bleached earth process, poorer color values are 20 reached, combined with unpleasant odor, a clearly higher NZ, a lower VI, a clearly poorer aging behavior, as well as inadequate removal of polycyclic aromatic hydrocarbons. Hydration methods, do, in fact, offer better yields with lower viscosity index (VI) and otherwise comparable values, but a quantitative removal of polycyclic aromatic hydrocarbons is possible only under extreme hydration conditions and employment of rare 25 metal catalysts, which is not customary in the practice of lubrication/base oil production. Not one of the currently known methods of reprocessing of waste oils into base oils, including the known distillation and extraction methods, is capable of removing the polycyclic aromatic hydrocarbons to the extent as it is possible with the here described method.

In the following, the invention is explained with the aid of examples, which shall not limit the scope of teaching according to the invention. To the person skilled in the art, additional specific embodiments are obvious within the scope of the inventive disclosure.

5 Example 1

Waste oil of category I according to Waste Oil V is distilled, while adding 0.5% of a 50% potassium hydroxide solution, in an apparatus, under vacuum of 600 mbar, in a temperature range of 140°C, for purposes of expelling water and the low boiling constituents.

10 The obtained dry oil, in a subsequent middle oil distillation is liberated, in a vacuum of 60 mbar and at a temperature in the sludge of the column of 260°C, from the middle distillate cut - boiling finish 380°C. The sludge product of the middle oil distillation column reaches a thin film evaporation unit, in which the non-destructive separation takes place, at a vacuum of 3 mbar and thermal carrier oil temperature of 384°C, of charged feed batch, into a lubricating oil distillate mixture and a bottom product. The lubricating oil distillate mixture is separated in a subsequent fractionation into boiling cuts, at process conditions of 80 mbar and 280°C distillation temperature. The thus obtained boiling cuts in viscosity state, at a viscosity of 40°C = 22 mm²/s and a viscosity of V40 at 38 mm²/s, are alternately extracted in a succeeding selective raffination with the solvent NMP. With a solvent oil ratio of 1.5:1, and an isothermal extraction temperature over the entire column path of 80°C, the employed oil (feed) is transported in counter flow to the utilized solvent NMP. During this process, the undesirable components, among others the polycyclic aromatic hydrocarbons dissolve from the charged feed, resulting, concurrently, in improvement in the quality of the charged lubricating oil distillate.

25 The raffinate NMP mixture leaving at the head of the column in accordance with the alternating operating mode, is subsequently transmitted to an NMP solvent medium reclaiming device, in order to once again employ said solvent in the process. The

attained lubricating oil distillate or lubricating oil raffinate of the respectively employed viscosity category V40, either 20 mm²/s or 36 mm²/s is subsequently used for the formulation of new lubricating oils, such as for example engine oils, transmission oils, hydraulic oils and other applications.

5 The resulting extract also passes through a solvent reclaiming plant in order to be able to reclaim the solvent NMP present in the extract for another new application. The resulting extract can be utilized as heating fuel oil or a fuel oil diluent in heating oil mixtures.

10 The bottom product from the thin film evaporation is subjected to a succeeding further thin film evaporation stage with a higher vacuum - 0.1 mbar and a temperature of 410°C. During this step there takes place a separation into a highly viscous lubricating oil fraction having a viscosity of 253 mm²/s and a remaining residue, which is used as heating oil mix-in component, for example as reduction oil in heating oil mixtures for the steel industry. The obtained highly viscous lubricating oil distillate is likewise 15 subjected to extraction with NMP in a succeeding selective raffination, with isothermal reaction conditions in the column at 90°C and a ratio of solvent: oil of 2:1. One obtains a qualitatively high grade highly viscous raffinate with a viscosity of 217 mm²/s and an extract which can also serve as mix in material for heating oil components or as heating oil itself for combustions, i.e. as charging material for generating heat or other purposes.

20 The obtained properties of the reclaimed base oil are as follows:

Table 1

| Raffinate | | |
|-------------------------------|----------------------|-------------|
| Temperature | [°C] | 80 isotherm |
| return oil phase from extract | | yes |
| NMP/Oil ratio | v/v | 1.5 |
| 5 Yield | [weight %] | 84 |
| Color ASTM | | 0.5 |
| Neutralizing Figure | [mg KOH/g] | 0.01 |
| Viscosity 40°C | [mm ² /s] | 20.93 |
| Viscosity 100°C | [mm ² /s] | 4.23 |
| 10 Viscosity Index | | 106 |
| Aromat Percentage CA (IR) | [%] | 3.5 |
| PAK, total n | [mg/kg] | 0.257 |
| Grimmer | | |
| 15 Benzopyrene | [mg/kg] | 0.0034 |

15 Example 2

The same procedure was followed as in Example 1, with the selected extraction conditions and the attained properties of the reclaimed base oils being represented in the Table below:

Table 2

| Raffinate | | |
|-------------------------------|----------------------|-------------|
| Temperature | [°C] | 80 isotherm |
| return oil phase from extract | | yes |
| NMP/Oil ratio | v/v | 1.8 |
| Yield | [weight %] | 85 |
| Color ASTM | | L 1.5 |
| Neutralizing Figure | [mg KOH/g] | <0.03 |
| Viscosity 40°C | [mm ² /s] | 36.05 |
| Viscosity 100°C | [mm ² /s] | 6.07 |
| Viscosity Index | | 114 |
| Aromat Percentage CA (IR) | [%] | 3.9 |
| PAK, total n | [mg/kg] | <1mg |
| Grimmer | | |
| Benzopyrene | [mg/kg] | * |

15 *was not ascertained

Example 3 to 5

The same procedure was following as in Example 1, with the selected extraction conditions and the attained properties of the reclaimed base oils being represented in Table 3 below:

| Table 3 | | | | |
|---------|-------------------------------|----------------------|----------------|-------------------------------------|
| | | Raffinate | | |
| 5 | Temperature | [°C] | 80 isotherm | 80 isotherm 80/25 gradient |
| | return oil phase from extract | | yes | yes no |
| | NMP/Oil ratio | v/v | 2.0 | 1.1 1.1 |
| 10 | Yield | [weight %] | 84 | 92 92 |
| | Color ASTM | | 1.0 | L 2.0 2.0 |
| | Neutralizing Figure | [mg KOH/g] | <0.01 | 0.03 0.04 |
| | Viscosity 40°C | [mm ² /s] | 36.00 | 36.44 37.03 |
| | Viscosity 100°C | [mm ² /s] | 6.08 | 6.07 6.10 |
| 15 | Viscosity Index | | 116 | 112 110 |
| | Aromat Percentage CA (IR) | [%] | 3.2 | 4.7 4.6 |
| | PAK, total n | [mg/kg] | 0.024 | 0.553 0.078 |
| | Grimmer | | | |
| 20 | Benzopyrene | [mg/kg] | 0.002 | 0.020 0.005 |

As is apparent from Table 3, it is possible to achieve excellent yields with both inventive process variations, i.e. with isothermal operation or with extraction with temperature gradients. The obtained base oils have, in addition, good color value, low neutralizing figures (NZ) and a high viscosity index (VI). The aromat percentage is in each instance clearly lowered, the contents of polycyclic aromatic hydrocarbons (PAK) lies far below 1 mg/kg and the contents of benzopyrene could be reduced to a range of less than 0.1 mg/kg. The contents of polychlorinated biphenylene (BCP) was below the detection limit of the analytical process. Consequently, the base oils attainable with the invention have an excellent quality.

1. Method for reprocessing waste oils and production of high-grade base oils, comprising the following steps:

5 A) distillation of the waste oil for removal of low boiling organic fractions, as well as drying of waste oil by removal of water;

 B) distillation under vacuum of the waste oil obtained according to step A) for segregation of fuel oil and diesel fractions with boiling cut of approximately 170 to 385°C in form of high grade fuel oils;

10 C) non-destructive distillation of the distillation residue from step B) by means of thin film evaporation in high vacuum to obtain a lubricating oil fraction with a standard viscosity range, which can be separated, as needed, via a subsequent distillative fractioning step - if appropriate under vacuum - into boiling cuts of different viscosity states;

15 D) if appropriate, non-destructive distillation of the bottom product from step C) in order to obtain a lubricating oil fraction of higher viscosity state from the higher boiling range which can be segregated, as needed, possible under vacuum, by means of a subsequent distillative fractioning step;

20 E) extraction of the fraction or fractions in the form of lubricating oil fractions or boiling cuts of different viscosity states from step C) and optionally D) with N-methyl-2-pyrrolidone (NMP) and/or N-formylmorpholine (NMF) as extraction medium in order to obtain very high grade base oils whereby the extraction is executed in such manner that undesirable constituents are removed in an almost quantitative manner, and the contents of the polycyclic aromatic hydrocarbons (PAK) and polychlorinated biphenyls (PCB) lies, respectively, clearly below 1 mg/kg.

2. Method according to Claim 1, characterized in that the waste oils to be reprocessed are treated with concentrated watery alkaline solution as reagent in order to segregate interfering constituents, in particular for process step C).

5 3. Method according to Claim 2, characterized in that the alkaline solution is added during distillation according to step A).

4. Method according to Claim 2 or 3, characterized in that the alkaline solution is potassium hydroxide solution.

10 5. Method according to Claim 4, characterized in that the alkaline solution is highly concentrated specifically approximately 5 to 50% potassium hydroxide solution.

6. Method according to Claim 5, characterized in that the feed (charge for extraction) is imparted an alkalinity reserve, which prevents the otherwise usual, partially even irreversible, acidulation of the reclaimed extraction medium.

15 7. Method according to Claim 1 or 2, characterized in that the distillation in step A) is performed at normal pressure or at slight under pressure of up to approximately 600 mbar and at a temperature of approximately 140 to 150°C.

8. Method according to at least one of the preceding Claims, characterized in that the extraction is performed in an extraction column, by counter flow process.

20 9. Method according to at least one of the preceding claims, characterized in that the extraction is performed in isothermal manner, at a temperature in the range of approximately 50 to 90°C.

10. Method according to Claim 9, characterized in that the extract phase is cooled down and that the settling oil phase is again added to the feed.

11. Method according to at least one of Claims 1 to 8, characterized in that the extraction is performed with a temperature gradient, whereby the temperature is 5 adjusted at the column head (run off raffinate) to approximately 50 to 90°C and at the column end (extract run-off) to approximately 10 to 50°C.

12. Method according to at least one of the preceding Claims, characterized in that the waste oil to be reprocessed has a contents of polychlorinated biphenyls (PCB) or PCB substitutes of up to approximately 250 mg/kg.

10 13. Method according to at least one of the preceding claims, characterized in that the waste oil to be reprocessed has a contents of vegetable oil(s) of up to approximately 5%.

14. Base oil, obtainable according to at least one of the method claims 1 to 13.

15 15. Use of the base oil according to Claim 14 as starter product for lubricants or for products of the petro-chemical industry.

Abstract

The invention relates to a method for reprocessing waste oils and producing high-grade base oils, whereby waste oil is treated by means of distillation, thin-film evaporation in a high vacuum, optional fractionation for separation into layers of different viscosities and subsequent extraction with N-methyl-2-pyrrolidone and/or N-formylmorpholine. The invention also relates to base oils that can be obtained by said method and to the use thereof. The inventive method is characterized in that approximately any waste oil can be used, including waste oils with polychlorinated biphenylene (PCB) or PCB substitutes with contents of up to 250 mg/kg. Furthermore, waste oils containing up to 5% vegetable oil can be present in the waste oil that is to be reprocessed without impairing the quality of the base oil. The undesirable constituents, especially polycyclic aromatic hydrocarbons and polychlorinated biphenylenes (PCB) or the substitutes thereof are removed in an almost quantitative manner.



Attorney Docket No. HMN 2 0016

DECLARATION AND POWER OF ATTORNEY FOR PATENT APPLICATION

As a below inventor, I hereby declare that:

My residence, post office address and citizenship are as stated below next to my name.

I believe I am the original, first and sole inventor(if only one name is listed below) or an original, first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought on the invention entitled:

METHOD FOR REPROCESSING WASTE OILS, BASE OILS OBTAINED ACCORDING TO SAID METHOD AND USE THEREOF

the specification of which
[] is attached hereto

was filed on May 3, 2001
Application Serial No. 09/831,104
and was amended on

(if applicable)

[] was filed as PCT on

I hereby state that I have reviewed and understand the contents of the above identified specification, including the claims, as amended by any amendment referred to above.

I acknowledge the duty to disclose information which is material to the examination of this application in accordance with Title 37, Code of Federal Regulations, ' 1.56 (a).

I hereby claim foreign priority benefits under Title 35, United States Code, ' 119 or 365(b) of any foreign application(s) for patent or inventor=s certificate, or 365(a) of any PCT international application which designated at least one country other than the United States of America, listed above and have also identified below, by check the box, any foreign application(s) for patent or inventor=s certificate, or of any PCT international application having a filing date before that of the application on which priority is claimed:

| | | |
|---------------------------|-----------|------------------------|
| Prior Foreign Application | | |
| 198 52 007.7 | Germany | 11/11/98 |
| (Number) | (Country) | (Day/Month/Year Filed) |

No
Certified Copy Attached?

I hereby claim the benefit under 35 U.S.C. 119(e) of an United States provisional application(s) listed below.

[] additional provisional application numbers are
Application No(s) (Day/Month/Year Filed) listed on a supplemental priority data sheet attached

I hereby claim the benefit under Title 35, United States, ' 120 of any United States application(s) or any PCT international application designating the United States of America, listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in the prior United States application or PCT International application in the manner provided by the first paragraph of Title 35, United States Code, ' 112, I acknowledge the duty to disclose material information which is material to patentability as defined in Title 37, of Federal Regulations Code, ' 1.56(a) which became available between the filing date of the prior application and the national or PCT international filing date of this application:

PCT/EP99/08667 11 November 1999

U.S. Parent Application
or PCT Parent Number

Parent Filing Date
(MM/DD/YYYY)

Parent Patent Number
(If Applicable)

Attorney Docket No. HMN 2 0016

POWER OF ATTORNEY: As a named inventor, I hereby appoint the following attorney(s) and/or agent(s) to prosecute this application and transact all business in the Patent and Trademark Office connected therewith.

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I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both under 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

1-00
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